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UCRL-PROC-233435

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August 7, 2007

15th APS Topical Conference on Shock Compression of Condensed Matter
Kohala Coast, HI, United States
June 24, 2007 through June 29, 2007

An X-ray Study of Shock-Recovered Tantalum Single Crystals

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Abstract: In this paper, we report shock-induced new grains and residual lattice tension in tantalum single crystals. The single crystals with orientations in [001], [011], [111], and [123] directions are shocked at ~ 55 GPa in gas gun under almost identical conditions. New grains in the shocked crystals are revealed by x-ray scanning analysis. Rather than lattice compression that is frequently probed by in situ x-ray diffraction technique, we find significant residual lattice tension in the recovered tantalum crystals. Such residual lattice tension is attributed to the dislocation cells and their deformation. The dislocation cells are accordingly estimated to be greater than 100 nm from broadening of x-ray diffraction peak.

Keywords: Tantalum, Single crystal, X-ray Diffraction, Shock, Lattice strain, Dislocations

INTRODUCTION

Tantalum and its alloys are known for their excellent ductility, workability and toughness. They have important applications under high pressures and high temperatures, as they are usually strong, hard and stable through wide temperature ranges. Up to date, numerous studies have been carried out to investigate their mechanical behavior at high strain rates and high pressures [1]. Most of them are focused on polycrystalline tantalum and alloys. But little is known about deformation and microstructure evolution of tantalum single crystals (TSC). We are studying shock-induced microstructure changes in TSC; the progress about crystals shocked at 55 GPa is reported in this paper.

EXPERIMENT

The disc-shaped single crystals are prepared using electro-discharged machining (EDM). The dimensions of the discs are ~ 2 mm thick and ~ 7.6 mm in diameter. Shock experiments are carried out in gas gun. Shot velocity is measured and pressure is estimated to be ~ 55 GPa. The simulation using a hydrodynamic code is carried out to check the wave profile. The simulated result is shown in Figure 1. The peak pressure at the sample center is ~ 55 GPa, consistent with the experimental measurement.

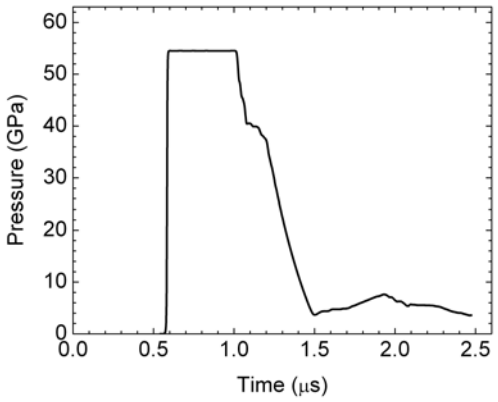


Figure 1: The wave profile in shock experiment.

After shock experiments, the shock recovered tantalum single crystals are ground and polished, then analyzed using x-ray diffraction. X-ray diffraction measurements are executed with a Philips PW-3070 diffractometer, while θ and 2θ are coupled. A power supply of 40 kV, 40 mA, and a Cu anode (λ = 1.54056 Å) with a Ni filter are used. The scan range is $2\theta = 20° - 130°$.

RESULTS

The x-ray diffraction spectrums of the shocked tantalum single crystals are shown in Figure 2. The strong primary peaks remain for all the shocked crystals, suggesting that the original crystal orientation of each crystal remains unchanged after shock. However, there are three new peaks in the x-ray diffraction spectrum of the shocked [001] crystal, namely (011), (112), and (013) peaks. According to the standard x-ray diffraction data of tantalum from the ICDD (International Center for Diffraction Data), intensity ratio for these three peaks should be 100:38:19. But the (013) peak is the strongest among the three. Similar observation is made for the shocked [011] crystal. According to the ICDD data, the new (002) peak should be weaker than the new (112) and (123) peaks. But actually the (002) peak are much stronger than the other two, indicating that the new grains have preferred orientations, leading to formation of texture. Four new peaks appear in the x-ray diffraction spectrum of the shocked [111] crystal. They are (011), (002), (112), and (013) peaks. Their intensity ratio follows the trend of the ICDD data. There is no new diffraction peak in the x-ray spectrum of the shocked [123] crystals.

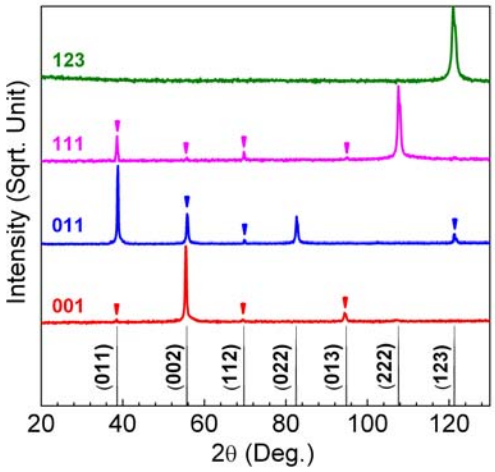


Figure 2: X-ray diffraction spectrums of the shocked crystals the four orientations. The additional peaks induced by shock loading are indicated by arrows. The permissible peaks and their indexes are also indicated at the bottom.

By comparing the primary x-ray diffraction peak positions of the shocked crystal and the corresponding pristine crystal of each orientation (Figure 3), we find significant peak shifting. The Brag angle of the (002) peak of the shocked [001] crystal is 27.76°, while that of the corresponding pristine crystal is 27.82°. The difference is - 0.06°. In other words, shock loading causes the primary (002) diffraction peak to shift ~ 0.06° toward a smaller value. Similar negative shifts also occur in crystals in all other orientations, as shown in Figures 3b-3d. The shifting magnitudes are -0.035°, -0.405°, and -0.160° for crystals in [011], [111], and [123] orientation, respectively.

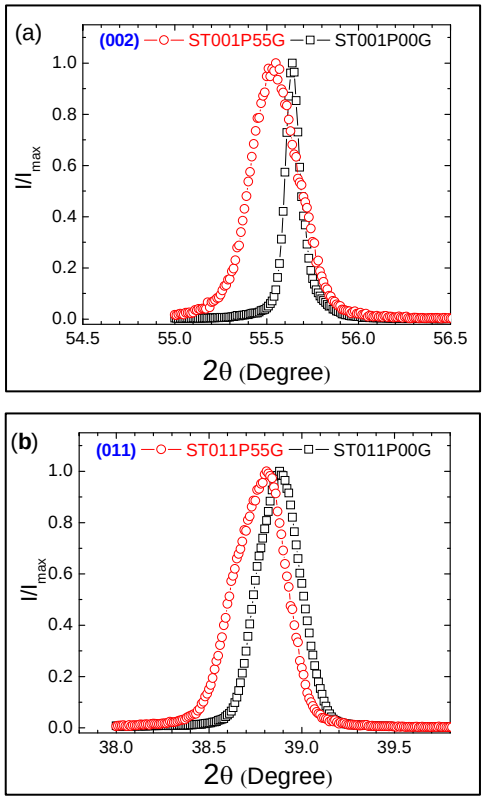
According to Bragg's Law, the product of interplanar lattice space d and the sine of Bragg angle, $\sin\theta$, is a constant, equal to one half of the x-ray wavelength, i.e. $\lambda = 2d\sin\theta$. A smaller Bragg angle corresponds to a larger interplanar lattice space. The change of the interplanar lattice space along the shock wave direction is estimated using the following formula: $\Delta d/d = \sin\theta_B^p / \sin\theta_B^s - 1$, in which θ_B^p and θ_B^s are the Bragg angles of the pristine and the corresponding shock-compressed crystals, respectively. The calculation gives a lattice stretch range between ~ 0.16% to ~ 0.20% for [001], [011], and [123] crystals. The lattice stretch in the shocked [111] crystal is up to 0.5% for reasons that are unknown.

DISCUSSION

It is well known that, a material under planar wave shock loading is typically subject to shock drive, release, and the following thermal recovery processes [2]. If the pressure is higher than the Hugoniot elastic Limits (HEL), the lattice is first elastically shock compressed, then plastically deformed leading to permanent microstructure changes and lattice reassembly [3-6]. Upon unloading, the lattice compression in metals at high pressure should be released upon unloading.

Tantalum single crystals in this study are shock loaded to a pressure of 55 GPa, which is at least ten times of the HELs for tantalum. The crystals are subject to significant plastic deformation and the subsequent release and

thermal recovery processes. The residual lattice tension in shock compressed tantalum crystals may be associated with permanent lattice and microstructure change induced by shock loading. Dislocation, dislocation cells, multiscale twins, and omega phase are the most likely microstructure features in shock loaded pure tantalum and tantalum alloys [7]. Among these features, dislocation cell structure is an important source to cause long-distance internal stresses, as it is a heterogeneous microstructure, consisting of cell wall with high-density dislocations and cell interior with low-density dislocations. Levine and his co-worker [8] have directly detected long-distance residual stresses/strains in the deformed lattices within individual dislocation cells, using an x-ray scanning probe with a beam size less than 1 micron. The residual strain is tension when the crystal is compressed, while it is compression when the crystal is stretched. This is a direct confirmation of a model prediction by Ungar, Mughrabi and others [9].



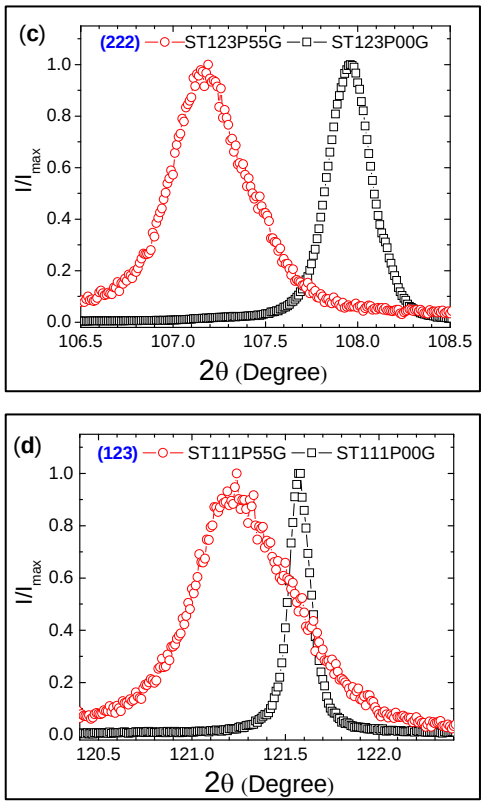


Figure 3: Shock-induced negative x-ray diffraction peak shifting and broadening for crystals in [001] (a), [011] (b), [111] (c), and [123] (d) orientations, respectively.

The residual lattice tension in our shock-loaded crystals is consistent with the abovementioned observations. This is indicative of the fact that dislocation cells are important in plastic deformation of shock loaded tantalum single crystals. They are formed during deformation, and then participate in the subsequent deformation before unloading. Dislocation cells are generally viewed as sub-grain boundaries, as the lattice orientation within each cell is slightly different from the neighboring ones. Thus, the sizes of dislocation cells can be estimated using the Scherer's Formula, i.e. $D_{cell} = 0.9\lambda / \beta \cos\theta_B$, in which D_{cell} is the average diameter of dislocation cells, β is the difference between the full-width at half maximum (FWHM) of the diffraction peaks of

the shock loaded and the corresponding pristine crystals. The estimated cell sizes are 128 nm for [001] crystal, 169 nm for [011] crystal, 167 nm for [111] crystal, and 116 nm for [123] crystal. All values are above 100 nm. The cell sizes are consistent with observations in shock loaded tantalum alloys [33], suggesting our earlier assumption is reasonable.

CONCLUSIVE REMARKS

New grains formed in the shock-recovered tantalum single crystals and revealed using x-ray diffraction analysis. The formation is orientation-dependent: new diffraction peaks appear in the spectrums of [001], [011], and [111] crystals, but not [123] crystal, although they are shock loaded at almost the same conditions.

The lattices in the recovered tantalum crystals are under significant residual tension, which is attributed to the dislocation cells formed during shock-induced deformation. The cell sizes are estimated to be larger than 100 nm for all crystals in all orientations. The estimated cell sizes are consistent with our experimental observations in other shocked tantalum.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48. The authors are grateful to Dr. Geoff Campbell for providing tantalum single crystal, to Mr. Sam Weaver for shock experiment.

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